LOW TEMPERATURE LIGNITE LIQUEFACTION

by

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Final Report

by

Saskatchewan Research Council

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ABSTRACT

Commonly lignite liquefaction is carried out at high pressure in the presence of carbon monoxide and hydrogen at temperatures ranging from 400°C to 460°C. Reaction time is usually less than one hour. In this investigation the reaction temperature is lowered to the 300°C to 400°C range while reaction time is extended several hours. It was found that the reaction rate decreases with temperature and that a long retention time will not compensate for the conversion loss due to low temperature.

INTRODUCTION

Transportation of coal as a slurry in a pipeline is one of the most economical methods of transportation in spite of high capital costs and a requirement for large amounts of water. Commercial size pipelines require a coal loading of 40 to 50 percent by weight of the total slurry, the remainder being water. This water presents problems as it may be scarce at the pipeline source, while at the delivery end it must be removed and treated before disposal.

One solution to the water consumption problem is to transport the coal in oil. Work at the Saskatchewan Research Council (1) and other agencies (2) has determined that coal-oil slurry transportation is feasible.

Oil is not necessarily found near coal fields but it may be manufactured from the coal itself using any one of a number of well established processes. Efficient coal liquefaction to oil requires powdered coal in the presence of oil, high temperature, high pressure, good mixing and a large amount of cooling water. The following table compares the two processes.

Process Condition	Slurry Pipelining	Liquefaction
Coal concentration (by vol.)	30% to 50%	30% to 50%
Coal Size (mesh)	14 to 200	30 to 200
Pressure (psig)	1200 to 1600	1000 to 4000
Temperature (°C)	15 to 20	400 to 500
Solvent or Carrier	water	oil
Residence Time	hours to days	minutes to hours

If water is to be eliminated as a carrier for coal, oil can be used, the oil can come from the coal itself. If water is to be eliminated as a coolant for liquefaction reactors, the surface area of the reactor has to be extended so that heat loss by natural convection and conduction to the environment is adequate. This extension can come from converting the reactor into a pipeline. Once the liquefaction is started, using a once only external charge of oil, part of the liquefaction product can be drawn off downstream and recycled to the entrance of the pipeline reactor and slurried with the incoming coal. In this way the pipelining of coal can be carried out in dry areas of the country without encroaching upon local water supplies.

Both processes share the drawbacks of high capital costs and high water consumption (3,4). If the two processes can be combined into one, the total capital cost can be reduced while the water demand can be virtually eliminated. This is the basic concept of the "Quol Process" for the simultaneous transportation and liquefaction of coal.* Because of the elimination of water usage in the Quol slurry pipelining concept, the energy density in the pipeline is doubled, resulting in much lower unit capital costs. Furthermore water acquisition and disposal costs are nil as well as the environmental impact of the huge water demand.

The pipeline reaction produces oil, gas and char which can be burned in a boiler to produce electrical power and process steam. Alternately the oil and gas can be farther processed while the char is burned to produce energy for the process.

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*Proposed by Quittenton Associates.

Very high temperatures are undesirable in a pipeline if high pressures are to be used. Due to potential long residence times in a pipeline, it may be possible to lower the reaction temperature from the 400-500°C range to perhaps 385°C in the initial recycle loop. Further downstream the reacting mixture will cool but the reaction might progress a considerable amount due to the fact that several hours to several days would pass before reaching its destination.

There are a number of steps in the overall process of simultaneous transportation and liquefaction of coal which require detailed examination. However, the concept of staged liquefaction at progressively lower temperatures and pressures over much longer time intervals is the key step. If high conversion of coal cannot be achieved under these conditions, the overall process likely would not be attractive economically. Therefore, the overall process should be investigated in two stages. The first stage should concentrate on liquefaction. If high conversions can be obtained under pipeline conditions then the second stage could be undertaken and would concentrate on the materials handling aspects.

It is proposed that a series of tests in a laboratory autoclave be undertaken for the first stage to determine the yields and reaction rates of the liquefaction step at various temperatures and pressures over periods up to 6 days. Lignite coal will be used as it is the coal in Western Canada which is closest to major markets in Ontario and Quebec. The recent work undertaken on Canadian lignite at the Université de Sherbrooke (5) provides a basis for these tests.

PROCEDURE

Lignite coal obtained from the Saskatchewan Power Corporations' Souris Valley mine was ground to 77% passing 200 mesh. This powdered coal was then reacted in a 2-litre stirred autoclave with hydrogen or a hydrogen/carbon monoxide mixture in the presence of a solvent made up of anthracene and tetralin. Reaction temperatures ranged from 300°C to 415°C and pressures between 8.8 and 17.1 M Pa. Reaction times varied between 20 minutes and several hours.

The solvent and produced oil were extracted in a Soxhlet extractor using toluene. The "overhead" gases were metered and analyzed in order to facilitate a mass balance.

Details of the autoclave and charging procedure are given in Appendix I.

RESULTS

The results are summarized in the "Table of Results" on page 6. Lignite conversions between 40 and 50 percent can be obtained at reaction temperatures between 360°C and 415°C. Extending the reaction beyond 20 minutes producing no significant increase in conversion. Specifically, reaction times of several hours at 300°C do not measurably increase yield.

Very little reaction occurs until the solvent/m.a.f. coal ratio is raised from 0.92 to 1.82 and one test indicates this value should be about 3.0.

The most favourable CO/H_2 molar ratio of the three used was a value of 3/1.

TABLE OF RESULTS

RUN NO.	DATE	MAXIMUM REACTION TEMP (°C)	TIME AT MAXIMUM TEMP (MIN)	TIME AT 300°C (MIN)	INITIAL PRESSURE MPa	MAXIMUM PRESSURE MPa	FINAL PRESSURE MPa	CO/H_2O MOLAR RATIO	SOLVENT TO MAF RATIO	ANTHRACENE TO TETRALIN RATIO	PERCENT CONVERSION	PERCENT SHORTAGE IN MASS BAL.
1	Jan 18/82	385	20	675	2.41	8.76	2.89	H_2 only	1.08	4.0	18.6	1.4
2	Jan 28/82	415	30	0	3.24	17.1	4.07	H_2 only	0.92	4.0	7.1	2.7
3	Feb 24/82	415	60	0	3.14	17.9	4.00	3/1	0.92	4.0	11.6	7.9
4	Mar 2/82	415	30	0	2.94	12.8	3.59	3/1	1.85	4.0	41.6	4.4
5	Blank Run	415	30	0	2.14	11.5	2.07	3/1	--	4.0	--	33.2
6	Apr 13/82	415	30	0	2.90	12.2	3.65	3/1	3.28	1.5	56.3	2.4
7	May 17/82	385	60	0	4.14	14.9	4.41	3/1	1.82	4.0	40.3	4.7
8	May 25/82	360	60	0	4.14	14.1	4.21	3/1	1.82	4.0	41.3	4.3
9	June 7/82	385	20	0	3.62	12.8	4.07	3/1	1.82	4.0	44.7	3.7
10	June 15/82	385	20	690	3.62	13.3	4.00	3/1	1.82	4.0	46.2	2.8
15	Aug 7/82	385	20	690	3.61	12.3	4.14	1/3	1.82	4.0	34.5	3.2
16	Aug 26/82	385	20	0	3.61	11.9	4.28	1/3	1.82	4.0	23.7	2.7
18	Sept 5/82	385	60	0	3.61	13.4	4.14	1/3	1.82	4.0	38.2	3.9

DISCUSSION OF RESULTS

A total of 13 autoclave tests were made on Saskatchewan lignite coal. The original intent was to vary temperature and reaction time and monitor percent conversion, however the solvent/m.a.f. coal ratio, the CO/H_2 molar ratio and the anthracene/tetralin ratio were also varied in order to optimize reaction conditions.

The following observations are presented with reference to the table of results on page 6 .

1. Comparing runs 1, 2, 3 and 4, no significant reaction occurred until the solvent to MAF ratio was raised to 1.85 from about 1.0. Raising the temperature, increasing the reaction time and going from straight hydrogen to a 3:1 ratio of CO to H_2 had no significant effect on product yield at the lower solvent to MAF ratio.
2. Run No. 5 was a blank run (no coal) to determine if any toluene insoluble compounds were forming from the anthracene-tetralin mixture at reaction conditions. None were found.
3. Comparing runs 4 and 6, an increase in the solvent to MAF ratio to 3.28 from 1.85 resulted in a yield increase to 56.3% from 41.6 percent. This increase may have been affected by the fact that the anthracene to tetralin ratio was lowered to 1.5 from 4.0.
4. In runs 7 and 8, an operating temperature of 385°C was compared to 360°C at 60 minutes duration. The two conversions, 40.3% and 41.3% are not significantly different, but comparing run 7 to run 9, time had a slightly inverse effect on percent conversion! Possibly there is an upper conversion limit at a given temperature which regresses with extended reaction time.

5. Comparing runs 9 and 10, only a slight increase in conversion is gained by operating for 11 1/2 hours at 300°C after the 20 minute reaction at 385°C. Conversion regression is not apparent here.
6. Comparing run 10 to run 15 and run 9 to run 16, a decrease in the CO/H_2 ratio from 3:1 to 1:3 significantly decreases the conversion. Comparing run 16 to run 18, an increase in reaction time at 385°C results in an increase in percent conversion at a CO/H_2 molar ratio of 1/3.
7. Runs 11 through 14 were scrapped due to leakage at the reactor closure gasket and thermocouple well and to difficulties with the temperature controller.

   Run number 17 was lost when an overheated oven ignited the extracted coal samples.

Different values for the solvent/m.a.f. lignite mass ratio were examined; 0.92, 1.82 and 3.25. At a value of 0.92 very little reaction occurred so the ratio was elevated to 1.82 where most of the cooks were carried out. One run at a ratio of 3.28 produced the best yield but this result was masked by a different anthracene to tetralin ratio. Fonseca et al. (5) used a solvent/m.a.f. ratio of 2.8.

Three values for the CO/H_2 molar ratio were tried; pure hydrogen, 1/3 and 3/1. A ratio of 3/1 was found to be best. Fonseca et al (5) found the ratio of 3/1 to be an optimum and suggested that CO acted as a scavenger for the oxygen in lignite as well as a contributor to the liquefaction process itself.

Using a solvent/m.a.f. ratio of 1.82 the slurry in the recirculation portion of a pipeline reactor would be about 36% solids by weight even if

no reaction occurred. This mixture is easily pipelined. However, at the end of the recirculation zone the recycle stream is removed leaving only the produced oil to act as the transport medium for the unreacted coal. The liquid phase must constitute about 50% of the total weight of the mixture for this to occur.

Using average values for coal analysis (Appendix II), a conversion of 46 percent of m.a.f. coal will provide this 50 percent slurry provided that the water in the coal remain liquid in the pipeline and none of the conversion is represented by gases. Unfortunately 46 percent conversion is the upper limit reached in this series of tests so higher conversions in the initial recirculation reactors are necessary to ensure that pipelining is feasible.

CONCLUSIONS

Lignite conversions in the range of 40 to 50% can be obtained at a reaction temperature of 385°C for a duration of 20 minutes. Extending the reaction time to 60 minutes at 385°C produces no significant improvement in conversion. Also, extending the reaction time to several hours at 300°C produces no measurable increase in conversion.

Fonseca (5) et al suggest that conversion reactions for lignite in the presence of carbon monoxide are rapid and that little is gained by an extended reaction time.

The minimum "conversion to oil" required to ensure that the reacted mixture can be slurry pipelined is about 46%. Reaction conditions used in this investigation are marginal and therefore must be optimized. A modest increase in temperature and in the solvent/m.a.f. lignite ratio would probably be adequate. Also, in future tests, anthracene oil should be used as a solvent instead of an anthracene-tetralin mixture. Problems with reactor cleaning and soxhlet extraction were encountered due to the high melting point of anthracene. The anthracene-tetralin solvent was suggested in the original Quol process.

RECOMMENDATIONS

A series of autoclave tests should be scheduled using the most effective solvent/m.a.f. ratio and CO/H_2 ratio found in this series. The temperature range should start at 385°C and terminate at about 420°C so that comparison can be made with this investigation. In future work anthracene oil should be used as a solvent and not anthracene-tetralin mixtures.

The following table can be used as a starting point for future work.

solvent----------anthracene oil

solvent/m.a.f.--------3/1

temperautre----------385 - 420°C

pressure----------10 - 17 MPa

CO/H_2 molar ratio-----3/1

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APPENDIX I

1. Equipment
2. Outline of Procedure
3. Sample Calculation For Charging Reactor

1. EQUIPMENT

AUTOCLAVE

The reaction vessel was a 2 litre high pressure agitated vessel manufactured by Pressure Products Industries Inc. of Warminster, Pennsylvania. Operating limits are 4000 psig at 385°C.

The reactor has a magnetically coupled agitator driven by a variable speed electric motor. The agitator has an electric tachometer.

The reactor is heated by a 2 K.W. electric heating mantle wrapped around the body of the reactor and controlled by a proportional controller sensing the temperature via a thermocouple inserted into a well dipping into the reacting mixture. A printing recorder coupled directly to the temperature controller records the temperature at one minute intervals.

The reactor has two calibrated pressure gauges, one 600 psig gauge for gas charging and one 5000 psig gauge for measurements at operating temperature.

EXTRACTION

The reaction product was exhaustively extracted with toluene in a 200 ml soxhlet extractor cup and dried to constant weight in an oven at 105°C.

VENTED GAS MEASUREMENT

The overhead (or vented) gas volume was measured using a "Precision" wet test meter. Glass sample bombs were used to collect samples for chromatographic analysis.

2. OUTLINE OF PROCEDURE

1. Weigh in coal, anthracene and tetralin to ± 0.01 g and seal reactor.
2. Pressure reactor up to 600 psig with nitrogen and let stand for 1-2 hours as a pressure test. Vent nitrogen to atmospheric pressure.
3. Add the two reaction gases, commencing with the lowest molar concentration gas first in order to facilitate more accurate addition.
4. Set agitator at 500 rpm.
5. Heat up to operating temperature using full power on the temperature controller.
6. Set data-logger to record temperature every minute. Record pressure at regular intervals.
7. At end of maximum temperature reaction, cool to second reaction temperature or to room temperature by passing water through internal cooling coils.
8. Vent reactor through gas meter recording operating temperature and pressure of the meter.
9. Open reactor, scrape out product from reactor and agitator and weigh to ± 0.01 g.
10. Soak agitator and inside of reactor overnight with toluene.
11. Soxhlet extract product, dry and weight thimbles to obtain extracted weight of solids.
12 Filter, dry and weigh sludge from reactor soak and add weight to above to obtain total extracted solids.

3. SAMPLE CALCULATION FOR CHARGING THE REACTOR

(Based on Run No. 10)

1. Weight of coal chosen was 225 g as is.

2. Coal analysis

Fixed C	36.2%	} MAF = 68.4%
Volatiles	32.2%	
Ash	15.0%	
H_2O	16.6%	
Total	100.0%	

3. MAF = 225 x 0.684 = 153.9 g

4. $\frac{\text{Solvent}}{\text{MAF}} = 1.83$ so Solvent = 1.83 x 153.9 = 281.6 g

5. $\frac{\text{Anthracene}}{\text{Tetralin}} = 4.0$ so Tetralin = $\frac{1}{5}$ x 281.6 = 56.3 g

Anthracene = $\frac{4}{5}$ x 281.6 = 225.3 g

6. Gas addition was such that the number of gram moles of CO + H_2 mixture was the same as the number of gram moles of H_2 added when 3% by weight on MAF of pure hydrogen was used.

i.e. $\frac{3}{100}$ x 153.9 = 4.62 gH_2 or 2.31 g moles of H_2.

But CO:H_2 was in the ratio of 3:1.

So H_2 added was 0.25 x 2.31 = 0.577 g. moles = 1.15 g.

CO added was 0.75 x 2.31 = 1.731 g. moles = 48.47 g.

7. Volume of coal added = 225 g /(1.65 g/cc) = 136 cc

Volume of anthracene added = 225.3 g /(1.25 g/ml) = 180 ml

Volume of tetralin added = 56.3g/(0.973 g/ml) = 58 ml

} 0.374 ℓ

Volume of reactor = 1.930 ℓ

Gas volume after solid and liquid reactants added = 1.930 - 0.374 = 1.556 ℓ

8. Nitrogen in reactor after nitrogen purge:

$$\text{g-moles of nitrogen} = \frac{0.95\ (\text{atm}) \times 1.556\ (\ell)}{0.0821\left(\frac{\ell\text{-atm}}{\text{g.mole}\ ^\circ\text{K}}\right) \times 293\ (^\circ\text{K})} = 0.0614 \text{ or } \underline{\underline{1.72\ \text{g}}}$$

9. Gas charging Pressures

$$\text{pressure of } (H_2+N_2) = \frac{(0.577 + 0.0614) \times .0821 \times 293}{1.556} = 9.86 \text{ atm.} = 145 \text{ psia}$$

$$= \underline{\underline{130 \text{ psig}}}$$

$$\text{pressure of } (CO+H_2+N_2) = \frac{(0.577+0.0614+1.731) \times .0821 \times 293}{1.556} = 36.6 \text{ atm}$$

$$= 538 \text{ psia} = \underline{\underline{523 \text{ psig}}}$$

APPENDIX II: Coal and Gas Analyses

Table 1 : COAL ANALYSIS

PROXIMATE ANALYSIS

		1	2	3	4
Fixed Carbon	(%)	29.8	36.4	36.0	36.9
Ash	(%)	12.4	15.0	15.0	15.1
Volatiles	(%)	26.4	32.4	32.0	33.2
H_2O	(%)	31.5	16.2	17.0	14.8
Total	(%)	100.0	100.0	100.0	100.0
MAF	(%)	56.2	68.8	68.0	70.1

ULTIMATE ANALYSIS

Carbon	(%)	41.6	42.4
H	(%)	3.1	2.8
N	(%)	1.1	0.9
S	(%)	0.32	0.38
O	(%)	10.0	23.6
Ash	(%)	12.4	15.1
H_2O	(%)	31.5	14.8
TOTAL	(%)	100.0	100.0
BTU		6803	

Table 2 TOLUENE EXTRACTION OF MOISTURE-FREE COAL

1. (-) 0.22% weight loss on extraction (weight gain).
2. 0.00% weight loss on extraction.

Table 3 SCREEN ANALYSIS OF GROUND COAL

Mesh	Weight percent
-50 + 100	0.08
+ 200	22.9
- 200	77.3
	100.0

Table 4 OVERHEAD GAS ANALYSES

(Volume Percent)

	Run #2	Run #3	Run #7	Run #8	Run #18
H_2	44	9.4	13.5	17	69
CO	0.8	40	7.1	10	9
CO_2	9	18	35.5	31	15
Methane	9.2	9.1	1.14	0.66	0.73
Ethane	6.5	1.3	1.4	0.44	0.21
Propane	5.4	0.8	0.98	0.36	0.25
Butane	2.2	0.35	0.29	0.10	0.08
Pentane	3.1	0.2	0.14	0.045	0.05
N_2 *	≃ 19	(20)	(38)	(40)	11.5
O_2 *	≃ 1	(1)	(2)	0.2	0.9
Total	100.2	100.35	100.05	99.805	106.72
Molecular Wt. Estimate	20.0	27.7	30.4	28.6	13.7
CO/H_2 input ratio	0/1	3/1	3/1	3/1	1/3

* Number in ( ) are estimates to facilitate calculation of molecular weight of overhead gas.

APPENDIX III: Reaction Material Balances

RUN NO. 1 DATE January 18/82

1. REACTOR CHARGE

coal: 175 g
Anthracene: 103.5 g
Tetralin: 25.9 g
H_2: 345 psig
CO: --

2. TEMPERATURE PROGRAM

Heat up to 385°C:	123 min.
Time at 385°C:	20 min.
Cool down to 300°C:	19 min.
Time at 300°C:	11 hr - 15 min.
Cool down to 25°C:	130 min.

3. PRESSURE PROFILE

Initial: 350 psig
Maximum: 1270 psig
Final: 420 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	119.7 g	Reactor Scrape out:	269.92 g
Ash:	26.2 g	Reactor Rinse estimate:	7.79 g
H_2O:	29.1 g	Overhead gas estimate:	27 g
Anthracene:	103.5 g	Total:	304.7 g
Tetralin:	25.9 g		
H_2:	2.9 g		
CO:	--		
N_2:	1.8 g		
Total:	309.1 g		

Mass loss: 309.1 - 304.7 = 4.4

% loss: $\frac{4.4}{309.1} \times 100 = 1.4$

5. OIL YIELD

Solids in: 119.7 + 26.2 = 145.9

Solids out - from extraction thimbles:	107.12 g
- filtration of extraction liquid:	13.04 g
- from reactor rinse:	3.47 g
- beaker wash & other:	--
Total solids out:	123.63 g

% Conversion: $\frac{(145.9 - 123.63)}{119.7} \times 100 = 18.6$

RUN NO. 2 DATE January 28/82

1. REACTOR CHARGE

coal: 250 g
Anthracene: 126 g
Tetralin: 31.5 g
H_2: 470 psig
CO: --

2. TEMPERATURE PROGRAM

Heat up to 415°C: 137 min.
Time at 415°C: 30 min.
Cool down to 25°C: 164 min.

3. PRESSURE PROFILE

Initial: 470 psig
Maximum: 2480 psig
Final: 590 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	171.0 g	Reactor Scrape out:	348.88 g
Ash:	37.5 g	Reactor Rinse estimate:	0.41 g
H_2O:	41.5 g	Overhead gas estimate:	53 g
Anthracene:	126.0 g	Total:	402.3 g
Tetralin:	31.5 g		
H_2:	4.2 g		
CO:	--		
N_2:	1.7 g		
Total:	413.4 g		

Mass loss: 413.4 - 402.3 = 11.1 g

% loss: $\frac{11.1}{413.4} \times 100 = 2.7$

5. OIL YIELD

Solids in: 171.0 + 37.5 = 208.5 g

Solids out - from extraction thimbles:	175.38 g
- filtration of extraction liquid:	20.76 g
- from reactor rinse:	0.23 g
- beaker wash & other:	--
Total solids out:	196.37 g

% Conversion: $\frac{(208.5 - 196.37)}{171.0} \times 100 = 7.1$

RUN NO. 3 DATE February 24/82

1. REACTOR CHARGE

coal: 250.0 g
Anthracene: 126 g
Tetralin: 31.5 g
CO+ H_2: 455 psig
CO: 342 psig

2. TEMPERATURE PROGRAM

Heat up to 415°C: 145 min.
Time at 415°C: 60 min.
Cool down to 25°C: 130 min.

3. PRESSURE PROFILE

Initial: 455 psig
Maximum: 2600 psig
Final: 580 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	171.0 g	Reactor Scrape out:	340.7 g
Ash:	37.5 g	Reactor Rinse estimate:	
H_2O:	41.5 g	Overhead gas estimate:	78 g
Anthracene:	126.0 g	Total:	418.5 g
Tetralin:	31.5 g		
H_2:	1.05 g		
CO:	44.1 g		
N_2:	1.8 g		
Total:	454.5 g		

Mass loss: 454.5 = 418.5 = 36 g

% loss: $\frac{36}{454.5} \times 100 = \underline{7.9}$

5. OIL YIELD

Solids in: 171.0 + 37.5 = 208.5 g

Solids out - from extraction thimbles:
- filtration of extraction liquid:
- from reactor rinse:
- beaker wash & other:

} 188.68g

Total solids out: 188.68g

% Conversion: $\frac{(208.5 - 188.68)}{171.0} \times 100 = 11.6$

RUN NO. 4 DATE March 2/82

1. REACTOR CHARGE

coal: 225 g
Anthracene: 227 g
Tetralin: 57 g
H_2: 96 psig
H_2+ CO: 426 psig

2. TEMPERATURE PROGRAM

Heat up to 415°C: 127 min.
Time at 415°C: 30 min.
Cool down to 25°C: 81 min.

3. PRESSURE PROFILE

Initial: 426 psig
Maximum: 1850 psig
Final: 520 psig

4. MATERIAL BALANCE

INPUT

MAF: 153.9 g
Ash: 33.7 g
H_2O: 37.4 g
Anthracene: 227 g
Tetralin: 57 g
H_2: 0.95 g
CO: 39.7 g
N_2: 1.6 g
Total: 551 g

OUTPUT

Reactor Scrape out: 453.09 g
Reactor Rinse estimate: 5.81 g
Overhead gas estimate: 68 g
Total: 527 g

Mass loss: 551 - 527 = 24 g

% loss: $\frac{24}{551} \times 100 = 4.4$

5. OIL YIELD

Solids in: 153.9 + 33.7 = 187.6 g

Solids out - from extraction thimbles: 94.68 g
- filtration of extraction liquid: 27.36 g
- from reactor rinse: 1.54 g
- beaker wash & other: --
Total solids out: 123.58 g

% Conversion: $\frac{(187.6 - 123.58)}{153.9} \times 100 = 41.6$

RUN NO. 5 DATE Blank Run

1. REACTOR CHARGE

coal: --
Anthracene: 127.0 g
Tetralin: 32.0 g
H_2: 93 psig
H_2+ CO: 311 psig

H_2O: 79.0 g

2. TEMPERATURE PROGRAM

Heat up to 415°C: 24 min.
Time at 415°C: 30 min.
Cool down to 25°C: 160 min.

3. PRESSURE PROFILE

Initial: 311 psig
Maximum: 1670 psig
Final: 300 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	0	Reactor Scrape out:	128.52 g
Ash:	0	Reactor Rinse estimate:	8.35 g
H_2O:	79.0 g	Overhead gas estimate:	25 g
Anthracene:	127.0 g	Water	28 g
Tetralin:	32.0 g	Total:	190 g
H_2:	1.0 g		
CO:	44.0 g		
N_2:	1.6		
Total:	284.6 g		

Mass loss: 284.6 - 190 = 94.6 g Reactor Leaked!

% loss: $\frac{94.6}{284.6} \times 100 = 33.2$

5. OIL YIELD

Solids in: 0

Solids out - from extraction thimbles: 0.06 g
- filtration of extraction liquid: --
- from reactor rinse: 0.04 g
- beaker wash & other: --
Total solids out: 0.10 g

% Conversion:

RUN NO. 6 DATE April 13/82

1. REACTOR CHARGE

coal: 200 g
Anthracene: 270 g
Tetralin: 180 g
H_2: 105 psig
H_2+ CO: 420 psig

2. TEMPERATURE PROGRAM

Heat up to 415°C: 116 min.
Time at 415°C: 30 min.
Cool down to 25°C: 128 min.

3. PRESSURE PROFILE

Initial: 420 psig
Maximum: 1770 psig
Final: 530 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	136.8 g	Reactor Scrape out:	596.30 g
Ash:	30.0 g	Reactor Rinse estimate:	6.24 g
H_2O:	33.2 g	Overhead gas estimate:	69 g
Anthracene:	270 g	Total:	672 g
Tetralin:	180 g		
H_2:	0.85 g		
CO:	35.62 g		
N_2:	1.7 g		
Total:	688.2 g		

Mass loss: 688.2 - 672 = 16.2 g

% loss: $\frac{16.2}{688.2} \times 100 = 2.4$

5. OIL YIELD

Solids in: 136.8 + 30.0 = 166.8 g

Solids out -		
- from extraction thimbles:	66.36 g
- filtration of extraction liquid:	22.48 g
- from reactor rinse:	0.93 g
- beaker wash & other:	--
Total solids out:	89.77 g

% Conversion: $\frac{(166.8 - 89.77)}{136.8} \times 100 = 56.3$

RUN NO. 7 DATE May 17/82

1. REACTOR CHARGE

coal: 250 g
Anthracene: 250 g
Tetralin: 62.6 g
H_2: 150 psig
H_2+ CO: 600 psig

2. TEMPERATURE PROGRAM

Heat up to 385°C: 117 min.
Time at 385°C: 60 min.
Cool down to 25°C: 40 min.

3. PRESSURE PROFILE

Initial: 600 psig
Maximum: 2160 psig
Final: 640 psig

4. MATERIAL BALANCE

INPUT

MAF: 171.0 g
Ash: 37.5 g
H_2O: 41.5 g
Anthracene: 250 g
Tetralin: 62.6 g
H_2: 1.28 g
CO: 53.8 g
N_2: 1.7 g
Total: 619.4 g

OUTPUT

Reactor Scrape out: 434.75 g
Reactor Rinse estimate: 69.62 g
Overhead gas estimate: 86 g
Total: 590.37 g

Mass loss: 619.4 - 590.37 = 29.03 g

% loss: $\frac{29.03}{619.4} \times 100 = 4.7$

5. OIL YIELD

Solids in: 171.0 + 37.5 = 208.5 g

Solids out - from extraction thimbles: 116.28 g
- filtration of extraction liquid: 5.52 g
- from reactor rinse: 17.79 g
- beaker wash & other: 0.03 g
Total solids out: 139.62 g

% Conversion: $\frac{(208.5 - 139.62)}{171.0} \times 100 = 40.3$

RUN NO. 8 DATE May 25/82

1. REACTOR CHARGE

coal: 250 g
Anthracene: 250 g
Tetralin: 62.6 g
H_2: 150 g
H_2+ CO: 600 psig

2. TEMPERATURE PROGRAM

Heat up to 360 °C: 116 min.
Time at 360°C: 60 min.
Cool down to 25°C: 37 min.

3. PRESSURE PROFILE

Initial: 600 psig
Maximum: 2050 psig
Final: 610 psig

4. MATERIAL BALANCE

INPUT

MAF: 171.0 g
Ash: 37.5 g
H_2O: 41.5 g
Anthracene: 250 g
Tetralin: 62.6 g
H_2: 1.28 g
CO: 53.8 g
N_2: 1.7 g
Total: 6.9.4 g

OUTPUT

Reactor Scrape out: 507.06 g
Reactor Rinse estimate: 1.13 g
Overhead gas estimate: 85 g
Total: 593 g

Mass loss: 619.4 - 593 = 26.4 g

% loss: $\frac{26.4}{619.4} \times 100 = 4.3$

5. OIL YIELD

Solids in: 171.0 + 37.5 = 208.5 g

Solids out - from extraction thimbles: 129.92 g
- filtration of extraction liquid: 7.44 g
- from reactor rinse: 0.31 g
- beaker wash & other: 0.27 g
Total solids out: 137.94 g

% Conversion: $\frac{(208.5 - 137.94)}{171.0} \times 100 = 41.3$

RUN NO. 9 DATE June 7/82

1. REACTOR CHARGE

coal: 225 g
Anthracene: 225 g
Tetralin: 56 g
H_2: 130 psig
H_2+ CO: 525 psig

2. TEMPERATURE PROGRAM

Heat up to 385°C: 119 min.
Time at 385°C: 20 min.
Cool down to 25°C: 59 min.

3. PRESSURE PROFILE

Initial: 525 psig
Maximum: 1860 psig
Final: 590 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	153.9 g	Reactor Scrape out:	454.10 g
Ash:	33.7 g	Reactor Rinse estimate:	--
H_2O:	37.4 g	Overhead gas estimate:	83 g
Anthracene:	225.0 g	Total:	537 g
Tetralin:	56.0 g		
H_2:	1.15 g		
CO:	48.5 g		
N_2:	1.7 g		
Total:	557.4 g		

Mass loss: 557.4 - 537 = 20.4 g

% loss: $\frac{20.4}{557.4} \times 100 = 3.7$

5. OIL YIELD

Solids in: 153.9 + 33.7 = 187.6 g

Solids out - from extraction thimbles: 109.46 g
- filtration of extraction liquid:
- from reactor rinse: } 9.43
- beaker wash & other:

Total solids out: 118.89 g

% Conversion: $\frac{(187.6 - 118.89)}{153.9} \times 100 = 44.7$

RUN NO. 10 DATE June 15/82

1. REACTOR CHARGE

coal:	225 g
Anthracene:	225 g
Tetralin:	56 g
H_2:	130 psig
H_2+ CO:	525 psig

2. TEMPERATURE PROGRAM

Heat up to 385°C:	119 min.
Time at 385°C:	20 min.
Cool to 300°C:	7 min.
Time at 300°C:	11 1/2 hr.

3. PRESSURE PROFILE

Initial:	525 psig
Maximum:	1930 psig
Final:	580 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	153.9 g	Reactor Scrape out:	452.31 g
Ash:	33.7 g	Reactor Rinse estimate:	7.40 g
H_2O:	37.4 g	Overhead gas estimate:	82 g
Anthracene:	225.0 g	Total:	542 g
Tetralin:	56.0 g		
H_2:	1.15 g		
CO:	48.5 g		
N_2:	1.7 g		
Total:	557.4 g		

Mass loss: 557.4 - 542 = 15.4 g

% loss: $\frac{154}{557.4} \times 100 = 2.8\%$

5. OIL YIELD

Solids in: 153.9 + 33.7 = 187.6 g

Solids out - from extraction thimbles:	107.93 g
- filtration of extraction liquid:	6.69 g
- from reactor rinse:	1.88 g
- beaker wash & other:	--
Total solids out:	116.50

% Conversion: $\frac{(187.6 - 116.50)}{153.9} \times 100 = 46.2$

RUN NO. 15 DATE August 17/82

1. REACTOR CHARGE

coal: 225 g
Anthracene: 225 g
Tetralin: 56 g
H_2 + CO: 524 psig
CO: 131 psig

2. TEMPERATURE PROGRAM

Heat up to 385°C:	108 min.
Time at 385°C:	20 min.
Cool to 300°C:	7 min.
Time at 300°C:	11 1/2 hrs.

3. PRESSURE PROFILE

Initial: 524 psig
Maximum: 1780 psig
Final: 600 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	153.9 g	Reactor Scrape out:	471.85 g
Ash:	33.7 g	Reactor Rinse estimate:	2.53 g
H_2O:	37.4 g	Overhead gas estimate:	36 g
Anthracene:	225 g	Total:	510.38 g
Tetralin:	56 g		
H_2:	3.5 g		
CO:	16.2 g		
N_2:	1.7 g		
Total:	527.4 g		

Mass loss: 527.4 - 510.38 = 17.02 g

% loss: $\frac{17.02}{527.4} \times 100 = 3.2$

5. OIL YIELD

Solids in: 153.9 + 33.7 = 187.60 g

Solids out - from extraction thimbles:	132.37 g
- filtration of extraction liquid:	1.31 g
- from reactor rinse:	0.75 g
- beaker wash & other:	0.11 g
Total solids out:	134.54 g

% Conversion: $\frac{(187.60 - 134.54)}{153.9} \times 100 = 34.5$

RUN NO. 16 DATE August 26/82

1. REACTOR CHARGE

coal: 225 g
Anthracene: 225 g
Tetralin: 56 g
H_2 + CO: 524 psig
CO: 131 psig

2. TEMPERATURE PROGRAM

Heat up to 385°C:	81 min.
At 385°C:	20 min.
Cool to 25°C:	63 min.

3. PRESSURE PROFILE

Initial: 524 psig
Maximum: 1730 psig
Final: 620 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	153.9 g	Reactor Scrape out:	475.56 g
Ash:	33.7 g	Reactor Rinse estimate:	1.76 g
H_2O:	37.4 g	Overhead gas estimate:	36 g
Anthracene:	225.0 g	Total:	513.32 g
Tetralin:	56.0 g		
H_2:	3.5 g		
CO:	16.2 g		
N_2:	1.7 g		
Total:	527.4 g		

Mass loss: 527.4 - 513.32 = 14.08 g
% loss: 2.7

5. OIL YIELD

Solids in: 153.9 + 33.7 = 187.6 g

Solids out - from extraction thimbles:	140.93 g
- filtration of extraction liquid:	1.92 g
- from reactor rinse:	0.53 g
- beaker wash & other:	0.07 g
Total solids out:	143.45 g

$$\% \text{ Conversion: } \frac{(187.6 - 143.45)}{153.9} \times 100 = 28.7$$

RUN NO. 18 DATE September 5/82

1. REACTOR CHARGE

coal: 225 g
Anthracene: 225 g
Tetralin: 56 g
H_2 + CO 524 psig
CO: 131 psig

2. TEMPERATURE PROGRAM

Heat up to 385°C: 85 min.
Time at 385°C: 60 min.
Cool to 25°C: 95 min.

3. PRESSURE PROFILE

Initial: 524 psig
Maximum: 1950 psig
Final: 600 psig

4. MATERIAL BALANCE

INPUT		OUTPUT	
MAF:	153.9 g	Reactor Scrape out:	467.63 g
Ash:	33.7 g	Reactor Rinse estimate:	3.44 g
H_2O:	37.4 g	Overhead gas estimate:	36 g
Anthracene:	225 g	Total:	507.07 g
Tetralin:	56 g		
H_2:	3.5 g		
CO:	16.2 g		
N_2:	1.7 g		
Total:	527.4 g		

Mass loss: 527.4 - 507.07 = 20.33 g
% loss: 3.9

5. OIL YIELD

Solids in: 153.9 + 33.7 = 187.6 g

Solids out - from extraction thimbles: 116.16 g
- filtration of extraction liquid: 11.71 g
- from reactor rinse: } 0.94 g
- beaker wash & other: }
Total solids out: 128.81 g

% Conversion: $\frac{(187.6 - 128.81)}{153.9} \times 100 = 38.2$